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IS 4737 (1982): Chromite for chemical industries [CHD 1 : Inorganic Chemicals]

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IS : 4737 - 1982

Indian Standard
SPECIFICATION FOR
CHROMITE FOR CHEMICAL INDUSTRIES
(First Revision)

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI .110002

Indian Standard
SPECIFICATION FOR
CHROMITE FOR CHEMICAL INDUSTRIES
(First Revision)

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Indian Standard
SPECIFICATION FOR
CHROMITE FOR CHEMICAL INDUSTRIES
(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 24 February 1982, after the draft finalized by the Inorganic Chemicals (Misc) Sectional Committee had been approved by the Chemical Division Council.

0.2 Chromite is the main chromium bearing mineral of commercial importance. The mineral chromite has the composition $\text{FeO} \cdot \text{Cr}_2\text{O}_3$, with a theoretical content of 68 percent Cr_2O_3 and 32 percent FeO by mass. It is a group of cubic minerals known as spinels which is represented by the general formula $\text{XO} \cdot \text{Y}_2\text{O}_3$ where 'X' is a divalent metal ion usually, iron, calcium, magnesium or manganese and Y is the trivalent metallic ion usually, iron, chromium or aluminium. In natural chromite deposits, replacement of chromium and iron by other elements usually occurs to some extent, chromium being replaced by aluminium and trivalent iron, and iron by magnesium. Consequently, commercial chrome ore rarely contains more than 50 to 55 percent chromium and some containing up to 20 percent of both Al_2O_3 and MgO .

0.3 Chromite finds extensive use in the metallurgical refractories and chemical industries, the respective use depending on the chemical composition. The chemical use of chromite is almost entirely in the primary production of sodium dichromate, and other chromium salts are subsequently produced.

0.4 As vanadium is present in chromite in small amount, it is counted as chromium in this standard, and no separate method has been given for the estimation of vanadium.

0.5 This standard was first published in 1968. It is being revised in the light of the experience gathered during testing. In this revision major changes have been effected in:

- a) The methods of determination of silica, alumina, chromium, iron, lime and magnesium; and
- b) The method for the preparation of sample.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for chromite for chemical industries.

2. REQUIREMENTS

2.1 Description — The material shall be either in the form of lumps or concentrates free from dirt and other foreign matter.

2.2 The material, when tested according to the methods prescribed in Appendix A, shall comply with the requirements specified in Table 1. Reference to the relevant clauses of Appendix A is given in col (4) of the table.

TABLE 1 REQUIREMENTS FOR CHROMITE FOR CHEMICAL INDUSTRIES

SL NO.	CHARACTERISTIC (ON DRY BASIS)	REQUIREMENT	METHOD OF TEST, REF TO CL NO. IN APPENDIX A
(1)	(2)	(3)	(4)
i)	Chromic oxide (as Cr_2O_3), percent by mass, <i>Min</i>	44·0	A-3
ii)	Total iron (as FeO), percent by mass, <i>Max</i>	20·0	A-3
iii)	Alumina (as Al_2O_3), percent by mass, <i>Max</i>	14·0	A-4
iv)	Silica (as SiO_2), percent by mass, <i>Max</i>	7·0	A-5
v)	Lime (as CaO), percent by mass, <i>Max</i>	3·0	A-5
vi)	Magnesia (as MgO), percent by mass, <i>Max</i>	14·0	A-5

3. PACKING AND MARKING

3.1 Packing — Unless otherwise agreed to between the purchaser and the supplier, the material shall be supplied in open wagons.

3.2 Marking — A good sized metallic or cardboard label bearing the following information with suitable paint or ink shall be conspicuously displayed on the carrier and also placed inside:

- a) Name of the material;
- b) Mass of the material;
- c) Lot number; and
- d) Manufacturer's name and recognized trade-mark, if any.

3.2.1 The material may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act, and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI Marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Marks may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 The method of drawing representative samples of the material shall be as prescribed in IS : 2109-1962*. However, the number of tests and criteria for conformity shall be as prescribed in **4.2** and **4.3** respectively.

4.2 Number of Tests — Tests for determining chromic oxide (as Cr_2O_3), shall be made on each of the individual samples separately. Tests for all other requirements shall be made on the composite sample only.

4.3 Criteria for Conformity

4.3.1 For Individual Samples — For chromic oxide tested on the individual samples, the mean and the range of test results shall be computed as follows:

$$\text{Mean } (\bar{X}) = \frac{\text{Sum of individual test results}}{\text{Number of test results}}$$

Range (R) = difference between the minimum and the maximum test results.

The lot shall be declared to be in conformity with chromic oxide requirement if the value of expression ($\bar{X} - 0.6 R$) is not less than the specified minimum value.

4.3.2 For Composite Sample — For declaring the conformity of the lot to the requirements tested on the composite sample, the test results in respect of each characteristic shall satisfy the corresponding specified requirements.

*Methods of sampling dolomite, limestone and other allied materials.

APPENDIX A (Clause 2.2)

ANALYSIS OF CHROMITE FOR CHEMICAL INDUSTRIES

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see IS : 1070-1977**) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. PREPARATION OF THE SAMPLE

A-2.1 Procedure — Crush the representative sample so as to pass through 150-micron IS Sieve. Powder about 10-15 g of the sample so prepared in an agate mortar with agate pestle till 100 percent of the sample pass through 63-micron IS Sieve. This sample should be taken for chemical analysis.

A-2.1.1 Free moisture in the sample may be determined separately on 1 g sample drying at $110 + 5^{\circ}\text{C}$ for 2 hours.

A-3. DETERMINATION OF CHROMIUM AND IRON

A-3.0 Outline of the Method — The sample is fused with sodium peroxide in nickel crucible. Chromium is converted to dichromate and estimated with the addition of excess ferrous ammonium sulphate in acidified solution which is back titrated with standard potassium permanganate solution.

A-3.1 Reagents

A-3.1.1 Sodium Peroxide — Powder.

A-3.1.2 Dilute Sulphuric Acid — 1 : 1 (*v/v*).

A-3.1.3 Ferrous Ammonium Sulphate Solution — 0.1 N.

A-3.1.4 Standard Potassium Permanganate Solution — 0.1 N.

A-3.1.5 Dilute Hydrochloric Acid — 1 : 1 (*v/v*).

A-3.1.6 Orthophosphoric acid

*Specification for water for general laboratory use (*second revision*).

A-3.2 Procedure — Weigh accurately 0·5 g of the prepared sample in a nickel crucible of about 30 ml capacity. Add and mix thoroughly with about 4 g of sodium peroxide with a dry glass rod. Heat the crucible over a flame till the mixture inside melts. Rotate the crucible gently with a pair of tongs to mix the contents well. Then keep the crucible over the flame at cherry red heat for about 5 minutes. Remove and allow the crucible to cool down. Introduce the crucible into a 400 ml beaker containing about 100-150 ml of water and covered with a watch glass. The crucible should submerge under water. Slowly boil till evolution of oxygen gas ceases and peroxide is destroyed. Remove the crucible and wash down the adhering solution and decomposed mass into the same beaker, taking help of policeman. Cool the solution to room temperature.

A-3.2.1 Filter through No. 40 Whatman filter paper or equivalent. Wash the residue with water till free from chromate. Dissolve the residue in hydrochloric acid of minimum quantity and precipitate with sodium peroxide. Boil off oxygen, filter and wash. Six to eight washings should be adequate. Combine the filtrates and transfer into 250 ml standard measuring flask and make up to volume.

A-3.2.2 Take 50 ml of the solution, acidify with dilute sulphuric acid and add 5 ml more. Add 50 ml of ferrous ammonium sulphate solution. Add 5 ml orthophosphoric acid and titrate against standard potassium permanganate solution to the grey end point. Carry out a blank titration 50 ml of the ferrous ammonium sulphate solution with standard potassium permanganate solution.

A-3.2.3 Calculation

Chromic oxide (as Cr_2O_3),

$$\text{percent by mass} = \frac{25 \cdot 32 \times N (V_1 - V_2)}{M}$$

where

N = normality of standard potassium permanganate solution,

V_1 = volume in ml of standard potassium permanganate solution required for the blank,

V_2 = volume in ml of standard potassium permanganate solution required for the titration, and

M = mass in g of the prepared sample taken for the test.

A-3.3 Treat the residue on the filter paper with hot dilute hydrochloric acid till entirely dissolved. Transfer and dilute to about 200 ml. Add 3-4 g of ammonium chloride. Heat to boil and add ammonia dropwise till alkaline. Boil for 4 to 5 minutes. Allow to settle. Filter through No. 41 Whatman filter paper or equivalent. Wash with hot 0·5 percent ammonium nitrate solution and finally twice with hot water.

A-3.3.1 Dissolve residue on filter paper in hot dilute hydrochloric acid and transfer into a beaker. Cool and transfer into a 250 ml standard measuring flask. Make up to volume.

A-3.4 Determination of Iron

A-3.4.0 Outline of the Method — Ferric iron is reduced with stannous chloride excess of which is oxidized by mercuric chloride. The reduced iron is titrated against standard potassium dichromate solution using sodium diphenylamine sulphonate solution as indicator.

A-3.4.1 Reagents

A-3.4.1.1 Stannous chloride solution — Dissolve by heating 60 g of pure stannous chloride in a mixture of 400 ml of concentrated hydrochloric acid and 600 ml of water until the solution is complete. Cool, add a few pieces of granulated tin and preserve the solution in an air-tight bottle.

A-3.4.1.2 Mercuric chloride solution — Prepare a saturated solution of mercuric chloride in water.

A-3.4.1.3 Sulphuric phosphoric acid mixture — Add slowly 150 ml of concentrated sulphuric acid to 700 ml of water with continuous stirring. Add to this solution 150 ml of syrupy phosphoric acid. Destroy any oxidizable impurities by adding potassium permanganate solution ($0\cdot1\text{ N}$) drop by drop until the pink colour of permanganate persists. Cool the solution.

A-3.4.1.4 Sodium diphenylamine sulphonate indicator solution — Dissolve 0·2 g of the reagent in 100 ml of hot water.

A-3.4.1.5 Standard potassium dichromate solution — 0·1 N. Dry pure potassium dichromate at $140 - 150^\circ\text{C}$ for one hour. Cool in a desiccator and transfer exactly 4·904 g to a one litre volumetric flask carefully through a funnel using water at room temperature. Wash the funnel thoroughly and dissolve the salt by shaking until the solution is complete. Make up to the mark and mix well.

A-3.4.2 Procedure — Take 100 ml from solution **A-3.3.1** in a 500 ml conical flask. Add 5 ml concentrated hydrochloric acid. Heat to boil. Add stannous chloride solution dropwise till the solution is colourless. Add two to three drops of stannous chloride in excess. Cool the solution quickly to room temperature.

A-3.4.3 Add 10 ml of mercuric chloride solution at a time and stir the solution. Silky white precipitate of mercurous chloride appears. Allow to stand for 5 minutes. Add 15 ml of sulphuric-phosphoric acid mixture. Dilute to about 200-250 ml with cold water. Add three to four drops of indicator solution. Titrate with standard dichromate solution till colour of the solution changes to stable blue violet.

A-3.4.4 Calculation

$$\text{Total iron (as Fe}_2\text{O}_3\text{), percent by mass} = \frac{A \times B \times 7.985}{C}$$

where

- A* = volume of standard potassium dichromate solution required,
- B* = normality of standard potassium dichromate solution, and
- C* = mass in g of the sample taken for the test.

A-4. DETERMINATION OF SILICA AND ALUMINA

A-4.0 General — The procedure involves decomposition of finely powdered chromite sample with perchloric acid. The decomposed material is taken up with dilute hydrochloric acid filtered and washed. Silica is determined by hydrofluorization. Hydroxides of iron, aluminium and chromium are then precipitated out with ammonia, filtered and washed. Calcium and magnesium are determined in the filtrate. Residue is redissolved in hydrochloric acid. Iron is precipitated with addition of sodium peroxide. Aluminium is determined from the filtrate after addition of ammonium nitrate.

A-4.0.1 Procedure — Take 0.25 g of finely agated sample (-0.63 micron IS Sieve) in a 250 ml beaker. Add 10 ml water and 25 ml of perchloric acid. Digest at low heat on a hot plate. Then most of black particles disappear, increase heat till copious fumes start (see Note). Extract with 20 ml dilute hydrochloric acid and digest. Filter through No. 42 Whatman filter paper transferring the entire residue and wash on filter paper with hot water till free from chloride. Collect the filtrate in a 400 ml beaker.

A-4.1 Determination of Silica

A-4.1.0 Outline of the Method — The residue is ignited and silica is determined by hydrofluorization in presence of 3-4 drops of sulphuric acid.

A-4.1.1 Reagents

A-4.1.1.1 Perchloric acid — 70 percent (*v/v*).

A-4.1.1.2 Hydrofluoric acid — 48 percent (*v/v*).

A-4.1.1.3 Dilute sulphuric acid — 18 N.

A-4.1.2 Procedure — Take the residue as in **A-4.0.1** in a previously ignited and weighed platinum crucible of about 25 ml capacity. Slowly burn off filter paper. Then ignite in a furnace up to a temperature of 1 000°C. Remove from the furnace, cool in a freshly charged desiccator and weigh. Repeat till mass is constant.

A-4.1.3 Add 3 to 4 drops of dilute sulphuric acid and 5 ml hydrofluoric acid. Heat on a hot plate till dry. Ignite slowly to 1 000°C and weigh. Repeat hydrofluorization till mass of the crucible is constant. Take final mass.

Fuse the residue with a little sodium carbonate, take up with dilute hydrochloric acid and mix with the filtrate as in **A-4.0.1**.

NOTE — In case black particles of undecomposed chromite persist, dilute with water, decant off the supernatant solution and retreat the particles with perchloric acid in the original beaker. Complete decomposition should not take more than 5 to 6 hours. Alternately, decompose 0.25 g of chromite by fusing with Na_2CO_3 (3 g), take up with dilute hydrochloric acid, evaporate and bake. Extract with 20 ml dilute HCl and proceed as in **A-4.0.1**.

A-4.1.4 Calculation

$$\text{Silica (as } \text{SiO}_2\text{), percent by mass} = \frac{M_2 - M_3}{M_1} \times 100$$

where

M_2 = mass in g of the residue obtained in **A-4.1.2**,

M_3 = mass in g of the residue obtained after hydrofluorization in **A-4.1.3**, and

M_1 = mass in g of sample taken for test in **A-4.0.1**.

A-4.2 Determination of Alumina

A-4.2.0 Outline of the Method — Alumina is determined by separating as sodium aluminate from the solution of mixed oxides and precipitating as aluminium hydroxide with ammonium nitrate and igniting to aluminium oxide.

A-4.2.1 Reagents

A-4.2.1.1 Dilute hydrochloric acid — 5 N.

A-4.2.1.2 Ammonium chloride — Solid.

A-4.2.1.3 Ammonium nitrate — Solid.

A-4.2.1.4 Dilute ammonium hydroxide — 3 N.

A-4.2.1.5 Dilute sodium hydroxide solution — 10 percent.

A-4.2.1.6 Sodium peroxide — Solid.

A-4.2.2 Procedure — Dilute the filtrate obtained in **A-4.0.1** to about 200 ml. Heat to boil after adding 4 g of ammonium chloride. Add with stirring dilute ammonium hydroxide till a faint ammoniacal smell is obtained. Boil for a few minutes. Allow the precipitate to settle, filter through No. 41 Whatman filter paper transferring bulk of the precipitate on the filter and wash with hot water 6 or 7 times. Dissolve the precipitate in the same beaker with dilute hydrochloric acid and repeat precipitation of hydroxides. Wash with hot water collecting the filtrate in the same beaker.

A-4.2.3 Take back the precipitate in the original beaker dissolving in hot dilute hydrochloric acid. Nearly neutralize with freshly prepared dilute sodium hydroxide solution. Add 1 to 2 g sodium peroxide. Dilute to about 200 ml. Heat to boil till evolution of oxygen ceases. Filter through No. 41 Whatman filter paper or equivalent. Repeat precipitation. Wash precipitate with hot water.

A-4.2.4 Treat the combined filtrate containing sodium aluminate and sodium chromate as in **A-4.2.3** with 4 to 5 g of ammonium nitrate with continuous stirring and precipitate aluminium hydroxide. Filter through No. 41 Whatman filter paper. Repeat precipitation. Wash precipitate on filter paper with hot 0.5 percent ammonium nitrate solution till free from chloride. Ignite precipitate on Al_2O_3 at about 1 000°C.

A-4.2.5 Calculation

$$\text{Alumina (as } \text{Al}_2\text{O}_3 \text{) percent by mass} = \frac{100 \times M_1}{M}$$

where

M_1 = mass in g of the ignited residue, and

M = mass in g of the material taken for test.

A-5. DETERMINATION OF LIME AND MAGNESIA

A-5.0 Outline of the Method — Combined filtrate as obtained in **A-4.2.2** is boiled down to less than 100 ml. Aliquots are taken for determination of calcium and magnesium complexometrically.

A-5.1 Reagents**A-5.1.1 Dilute Hydrochloric Acid — 5 N.**

A-5.1.2 Potassium Hydroxide Solution — approximately 10 N. The solution should be freshly prepared from AR quality potassium hydroxide.

A-5.1.3 Buffer Solution (pH 10) — Dissolve 70 g of ammonium chloride in 200 ml of water, add 570 ml of concentrated ammonia (density 0·88 - 0·90) and dilute to 1 litre.

A-5.1.4 Patton and Reeder's Indicator — Mix 0·1 g of 2 hydroxy 1-(2-hydroxy-4-sulpho-1-naphthalzo)-3-naphthoic acid with 10 g of potassium chloride and grind into a fine mixture in a glass mortar. Preserve in a well stoppered dry amber coloured glass bottle.

A-5.1.5 Erio Chrome Black T Indicator — Dissolve 0·5 g of Erio Chrome Black T indicator in 100 ml of rectified spirit and preserve in amber coloured glass bottle. This indicator should be freshly prepared preferably.

A-5.1.6 Standard EDTA Solution — Weigh 7·444 g of disodium ethylene diamine tetracetate dihydrate (EDTA) and dissolve in deionized water. Make up the volume to one litre. Mix thoroughly. The solution is 0·02 M.

A-5.1.7 Potassium Cyanide and Hydroxylamine Hydrochloride Pure

A-5.1.8 Deionized Water

A-5.2 Determination of Calcium Oxide

A-5.2.1 Procedure — The combined filtrate as in **A-4.2.2** is boiled down to less than 100 ml cooled and made up to volume 100 ml in a standard measuring flask. Take two portions of 25 ml each in two conical flask or white porcelain dish of 100 ml capacity. Add about 5 ml of potassium hydroxide solution to one flask. Add about 0·2 to 0·4 g P and R indicator and quickly titrate till the colour changed to violet blue. Note the volume of EDTA consumed. Add the same EDTA volume less 1·0 ml directly to the second flask. Add 5 ml of potassium hydroxide solution and about 50 mg each of potassium cyanide and hydroxylamine hydrochloride. Add 0·4 g indicator and immediately titrate with continuous stirring until no further colour changes and the test solution is clear blue without any tinge of violet. Volume of EDTA consumed in the final titration should be taken for calculating CaO percent (**A-5.2.2**).

A-5.2.2 Calculation

$$\text{Calcium oxide (as CaO), percent by mass} = \frac{0\cdot11216 V_1}{M}$$

where

V_1 = volume in ml of 0·02 M EDTA solution required for final titration, and

M = mass in g of the material contained in the aliquot of solution taken for the titration.

A-5.3 Determination of Magnesia

A-5.3.1 Procedure — Take 25 ml aliquot from **A-5.1.2** in a white porcelain dish of 100 ml capacity. Add 25 ml of ammonium chloride and ammonium hydroxide buffer solution and a few drops of Eriochrome Black T and titrate with EDTA solution till the colour changes from wine red to indicator blue.

A-5.3.2 Calculation

$$\text{Magnesium (as MgO), percent by mass} = 0.08062 \times \frac{V_2 - V_1}{M}$$

where

V_2 = volume of 0.02 M EDTA solution required for the titration,

V_1 = volume of 0.02 M EDTA solution required for the sample solution, and

M = mass in g of the material contained in the aliquot of solution taken for the titration.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	Unit	Symbol
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	Unit	Symbol
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	Unit	Symbol	Definition
Force	newton	N	1 N = 1 kg.m/s ²
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m ²
Frequency	hertz	Hz	1 Hz = 1 c/s (s ⁻¹)
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m ²

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KANPUR 208005	4 72 92
PATNA 800013	6 28 08
TRIVANDRUM 695001	32 27